Section VII: Identification of Opiates

I. Introduction:

Opiates and its derivatives are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. However, a preliminary analysis is performed using the computerized Identidex Imprint Identification program within the Micromedex Healthcare Series or similar web based program. The samples are extracted by a simple solvent extraction procedure. However, extraction by The ANOR procedure can also be used (See Section X).

II. Reagents:

A.) Petroleum ether
B.) Methanol (GC solvent rinse)
C.) 9:1 Methylene Chloride/NH_4OH

III. Equipment:

A.) Analytical balance
B.) Magnifying microscope or magnifying glass
C.) 2 mL autosampler vials with Teflon caps
D.) GC/FID: HP 6890 or 7890A series
E.) GC/MS: HP 6890/5973 or HP 7890A/5975C series
F.) Computer with Identidex Imprint Identification program.

IV. Procedure:

A.) Imprint Identification

1. Observe any imprint on tablet or capsule samples. Use a microscope or magnifying glass if necessary.
2. Record actual imprint, color, and shape of tablets or capsules in logbook.
3. On computer with Micromedex Healthcare Series, log onto the Identidex Imprint Identification page.
4. Enter the imprint code.
5. The program will access the database.
6. The identification, description and classification of the drug will appear. Read and verify that the computer's description matches your sample's description.
7. Print out the results, record results in logbook and file with the sample paperwork.

B.) Chromatography by GC/FID and GC/MS

1. If capsule or tablet contains no identifiable imprint code or if sample is in a powder form, the sample must be analyzed by GC/FID and GC/MS.
2. Place ¼ to ½ of tablet/capsule or 5 mg of powder sample into a 2 mL autosampler vial.
3. Add 1-2 mL of 9:1 Methylene Chloride/NH_4OH to vial and cap.
4. Place on GC/FID autosampler and run with regular sequence (STD, BLK, Samples, STD).
5. GC/FID conditions are as follows:

Method: EXP.M

Oven:

Initial Temp: 245°C
Initial Time: 0.00 min.
Rate: 10°/min.
Final Temp: 290°C
Run Time: 10 min.
Max. Temp: 325°C
Equilibration Time: 0.5 min.

Inlet:

Mode: split (35:1)
Initial Temp: 250°C
Pressure: 24.99 psi
Gas Type: Helium

Column:

Capillary: HP-1 30m x 320um
Initial Flow: 3.3 mL/min.

Detector:

Temp: 300°C
Hydrogen Flow: 30.0 mL/min.
Air Flow: 400 mL/min.
Makeup Gas: Helium

6. Obtain chromatographs. If sample contains any opiates or opiate derivatives the instrument will detect a total ion peak with a retention time characteristic of that compound and will generate a report with accompanying chromatograph.
7. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
8. Place the same sequence that ran on the GC/FID on the GC/MS autosampler.
9. GC/MS conditions are as follows:

Method: EXP.M

Oven:

Initial Temp: 230°C
Initial Time: 0.00 min.
Max. Temp: 325°C

Equilibration Time: 0.50 min.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 10 min.

Inlet:

Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium

Column:

Capillary: HP-1MS 25m x 200um x 0.33um
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.

10.) If opiates are present in sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity if the peak and its ion abundance.

V. Results:

A.) For the Identidex Imprint Identification procedure, record the identity of the sample in the logbook, as well as on the evidence cards that came with the samples. Be sure to include date of analysis, results, and initials. Also, print out the results from the computer and file it with that sample number's paperwork.

B.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.

C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.